Studies on Acetylene and its Derivatives. (X) : Studies on Polyvinylidenechloride (1) : Preparation of the Monomer

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24. Studies on Acetylene and its Derivatives. (X)

Studies on Polyvinylidenechloride (1)
Preparation of the Monomer.

Sango Kunichika and *Susumu Hirase.*

Vinylidenechloride was prepared by the dehydrochlorination of 1,1,2-trichloroethane, which had been obtained by the action of chlorine in the liquid phase (using trichloroethane as a solvent) upon vinylchloride contaihing 10–20 vol. % of acetylene. The results obtained on each reaction are as follow:

A) Trichloroethane

1. The higher the reaction temperature and also the higher the mol ratio of chlorine for vinylchloride, the more the formation of higher boiling products. The best yield (ca. 90%) of trichloroethane is obtained at lower temperature (0–20°), when the mol ratio of chlorine for vinylchloride is 1.2 : 1.

2. The rate of flow of the mixed gases and the height of the liquid layer do not largely effect on the yield.

3. Explosion, due to the presence of acetylene, does not occur when the content of vinylchloride is more than 70 vol. % in the gas.

B) Vinylidenechloride

1. Trichloroethane is easily dehydrochlorinated to vinylidenechloride in a yield of about 70% by the action of a slight excess of sodium-hydroxide solution (3.5–4%). When the concentration of sodium-hydroxide is higher, the reaction becomes difficult because of decrease of solubility of trichlorethane.

2. Vinylidenechloride is also obtained in a yield of 88% by using milk of lime as a dehydrochlorinating agent.

25. Alkylation of Dioxybenzenes by p-Toluolsulfonic Acid Ester.

Ryuzaburo Nodzu, Sango Kunichika and *Sinsaburo Oka.*

To study the bacteriostatic action on Mycobacterium tuberculosis, dioxybenzene monoalkyl ethers were prepared. As alkylating agent *p*-toluolsulfonic acid ester was used. Methyl, ethyl and butyl esters were prepared by adding 5N-sodium hydroxyde to a mixture of alcohol and *p*-toluolsulfonyl chloride with the yield: methyl ester 85%, ethyl ester 70% and butyl ester 56%.

Although butyl ester had been obtained by using pyridin instead of sodium hydroxyde aqueous solution we obtained it by using γ-picoline (B.P. 143–147°) with the yield: 85%. Hexyl ester was prepared also in an almost similar manner with the yield: 82%. Alklation was quite easily carried on as follows; 0.1 mol natrium